

FACILITY FORM 602

N 66-17436

(ACCESSION NUMBER)

(THRU)

46

(PAGES)

(CODE)

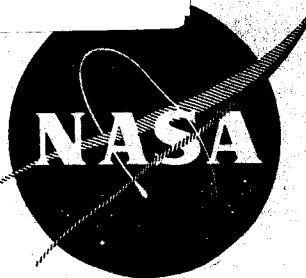
15

(CATEGORY)

CR-54891

(NASA CR OR TMX OR AD NUMBER)

NASA CR-59
FWA-2750



GPO PRICE \$

CFSTI PRICE(S) \$

Hard copy (HC) 2.00

Microfiche (MF) .50

653 July 65

SEMI-ANNUAL PROGRESS REPORT
DETERMINATION OF THE EMISSIVITY OF MATERIALS

by
W. Luoma
R. C. Emanuelson

prepared for
National Aeronautics and Space Administration
Contract NAS3-4174

Pratt & Whitney Aircraft DIVISION OF UNITED AIRCRAFT CORPORATION

U
A

EAST HARTFORD • CONNECTICUT

NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

- A.) Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
- B.) Assumes any liabilities with respect to the use of or for damages resulting from the use of any information, apparatus, method or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with NASA, or his employment with such contractor.

Requests for copies of this report
should be referred to:

National Aeronautics and Space Administration
Office of Scientific and Technical Information
Washington 25, D.C.
Attention: AFSS-A

Semiannual Progress Report
Determination of the Emissivity of Materials
Contract NAS3-4174

Report Period: May 15, 1965 through November 14, 1965

Technical Management: National Aeronautics and
Space Administration, Lewis Research Center
Space Power Systems Division
Robert L. Davies

December 20, 1965



Written by:

W. Luoma
W. Luoma
Analytical Engineer

Approved by:

R. C. Emanuelson
R. C. Emanuelson
Program Manager

W. J. Lueckel
W. J. Lueckel, Chief,
Space Power Systems

Pratt & Whitney Aircraft

DIVISION OF UNITED AIRCRAFT CORPORATION

U
A

E A S T H A R T F O R D • C O N N E C T I C U T

FOREWORD

This report describes the research activity carried out in fulfillment of Contract NAS3-4174 during the period from May 15, 1965, through November 14, 1965. Contributors to this report included G. Mikk and W. Atkinson. The work was conducted under the direction of the Space Power Systems Division, Lewis Research Center, National Aeronautics and Space Administration, with Robert L. Davies as Project Manager.

ABSTRACT

N66 17434

The testing of coatings applied to AISI-310 stainless steel and columbium-1 percent zirconium was continued during the report period. In addition, a study of the adherence, compatibility, and emittance of selected materials applied to beryllium was initiated.

A columbium-1 percent zirconium tube coated with iron titanate completed 10,000 hours of testing at 1700°F. During the test, the emittance decreased slightly from 0.88 to 0.84, with most of the decrease occurring during the first 1800 hours. The specimen is being subjected to a thorough analysis including spectrographic, X-ray diffraction, vacuum fusion, microhardness, and microprobe analyses. On the basis of emittance testing and supplementary adherence testing, it appears that this material combination is suitable for space radiator use at 1700°F. Analyses to determine the compatibility of the materials have not yet been completed.

An AISI-310 stainless steel tube coated with calcium titanate completed 10,600 hours of testing at 1350°F during the report period. The emittance of this specimen has decreased from 0.91 to 0.89. On the basis of the emittance test and on supplementary adherence testing conducted during the last six-month period, it appears that this material combination is suitable for space radiator use at 1350°F.

An AISI-310 stainless steel tube coated with iron titanate has been tested at 1350°F for 9,600 hours. The emittance has been very stable, ranging only between 0.89 and 0.88. This material combination also appears to be suitable for space radiator use at 1350°F.

A columbium-1 percent zirconium tube coated with aluminum oxide-aluminum titanate has been tested for 5,300 hours at 1700°F. The emittance of this specimen, however, has decreased steadily from 0.87 to 0.82. If it continues to decrease, the test will be terminated.

Aging tests were conducted on beryllium specimens with coatings of calcium titanate and iron titanate. The calcium-titanate coated specimens were aged for 100 hours and for 500 hours at 800°F. The post-test analyses for these specimens have not been completed, but preliminary results indicate that aging produced no significant

changes. The iron-titanate coated specimens were aged for 100 hours at 800°F. Preliminary results from post-test analyses for these specimens also reveal no significant changes, although a small amount of diffusion across the coating-substrate interface may have occurred.

A beryllium strip coated with iron titanate was emittance tested for 670 hours at temperatures from 800°F to 1100°F. The emittance was between 0.89 and 0.90 throughout the test.

auth or

TABLE OF CONTENTS

	<u>Page</u>
Foreword	ii
Abstract	iii
List of Figures	vi
List of Tables	viii
I. Introduction	1
II. Background	1
III. Long-Term Emittance Testing	2
A. Introduction	2
B. Iron Titanate on Columbium-1 Percent Zirconium	2
C. Calcium Titanate on AISI-310 Stainless Steel	4
D. Iron Titanate on AISI-310 Stainless Steel	5
E. Aluminum Oxide-Aluminum Titanate on Columbium-1 Percent Zirconium	5
IV. Beryllium Studies	7
A. Introduction	7
B. Aging Tests	7
1. Apparatus	7
2. Specimen Preparation	8
3. Test Procedure	10
4. Test Results	11
a. Calcium-Titanate-Coated Beryllium Specimens	11
b. Iron-Titanate-Coated Beryllium Specimens	13
C. Emittance Tests	15
1. Introduction	15
2. Test Results	15
V. Conclusions	16
VI. Future Work	17

LIST OF FIGURES

<u>Number</u>	<u>Title</u>	<u>Page</u>
1	Iron-Titanate-Coated Columbium-1 Percent Zirconium Specimen at 1700°F After 10,000 Hours of Testing	18
2	Pressure and Total Hemispherical Emittance for Iron-Titanate-Coated Columbium-1 Percent Zirconium Specimen	19
3	Iron-Titanate-Coated Columbium-1 Percent Zirconium Specimen After 10,000 Hours of Testing at 1700°F	20
4	Photomicrograph of Iron-Titanate on Columbium-1 Percent Zirconium After 10,000 Hours of Testing at 1700°F	21
5	Pressure and Total Hemispherical Emittance for Calcium-Titanate-Coated AISI-310 Stainless Steel Specimen	22
6	Calcium-Titanate-Coated AISI-310 Stainless Steel Specimen After 10,000 Hours of Testing at 1350°F	23
7	Pressure and Total Hemispherical Emittance for Iron-Titanate on AISI-310 Stainless Steel	24
8	Iron-Titanate-Coated AISI-310 Stainless Steel Specimen After 9000 Hours of Testing at 1350°F	25
9	Pressure and Total Hemispherical Emittance for Aluminum Oxide-Aluminum Titanate-Coated Columbium-1 Percent Zirconium	26

LIST OF FIGURES (Cont'd)

<u>Number</u>	<u>Title</u>	<u>Page</u>
10	Aluminum Oxide-Aluminum Titanate-Coated Columbium-1 Percent Zirconium Specimen After 4823 Hours of Testing at 1700°F	27
11	Vacuum Aging Furnaces Used for Beryllium Studies	28
12	Control Console for Beryllium Aging Studies	29
13	Appearance of Calcium Titanate-Coated Beryllium Specimen Before Aging, After Aging for 100 Hours at 800°F, and After Aging for 500 Hours at 800°F	30
14	Appearance of Iron-Titanate-Coated Beryllium Specimen Before Aging and After Aging for 100 Hours at 800°F	31
15	Total Hemispherical Emittance of Iron- Titanate-Coated Beryllium	32
16	Iron-Titanate-Coated Beryllium Specimen After Emittance Testing at Temperatures from 800°F to 1100°F	33

LIST OF TABLES

<u>Number</u>	<u>Title</u>	<u>Page</u>
1	Spectrographic Analysis Results for Iron-Titanate Coating on Columbium-1 Percent Zirconium Tube	3
2	Microhardness Results for Columbium-1 Percent Zirconium Tube Coated with Iron Titanate and Tested at 1700°F for 10,000 Hours	4
3	Microhardness Results for Beryllium Plates (as Received) Used in Aging Studies	9
4	Particle Size Distribution of Iron Titanate Powder Used for Beryllium Studies	10
5	Weights of Calcium-Titanate-Coated Beryllium Specimens Before and After Testing at 800°F	11
6	Spectrographic Analysis Results for Calcium-Titanate and Iron-Titanate Applied to Beryllium Substrates	12
7	Microhardness Results for Beryllium Plate Coated With Calcium-Titanate and Aged for 500 Hours at 800°F	13
8	Weights of Iron-Titanate-Coated Beryllium Specimens Before and After Testing at 800°F for 100 Hours	14
9	Microhardness Results for Beryllium Plate Coated With Iron-Titanate	14

I. INTRODUCTION

A program is being conducted to determine the suitability of selected high-emittance materials for use as coatings on nuclear space power-plant radiators. The program is divided into two phases. In one phase, selected coatings are being evaluated for emittance stability, adherence, and compatibility when applied to AISI-310 stainless steel or columbium-1 percent zirconium. These tests are being conducted for periods of at least 10,000 hours at temperatures of 1350°F and 1700°F.

In the second phase, selected coating materials are being applied to beryllium and evaluated for emittance stability, adherence, and chemical and metallurgical changes when exposed to temperatures of 800 or 1400°F for periods up to 1000 hours. This phase was initiated during the current report period.

II. BACKGROUND

During the first twelve months of the program, seven materials were screened for long-term emittance testing. Four coating-substrate combinations appeared to be suitable: iron titanate on AISI-310 stainless steel, iron titanate on columbium-1 percent zirconium, calcium titanate on AISI-310 stainless steel, and a stabilized titanium oxide composition on columbium-1 percent zirconium. The stabilized titanium oxide coating deteriorated rapidly during the first 100 hours of long-term testing, however, and the test was terminated. Subsequently, a long-term test was initiated for a columbium-1 percent zirconium tube coated with aluminum oxide-aluminum titanate. Adherence tests were conducted for iron-titanate coatings and the bond strength to their respective substrate materials was found to be excellent.

III. LONG-TERM EMITTANCE TESTING

A. Introduction

The long-term total hemispherical emittance testing of four coated specimens was continued during the report period. Originally, the tests were scheduled to be terminated after 5000 hours of continuous testing. However, in May 1965, the contract was extended and the test period was increased to 10,000 hours. By the end of the report period, one of the specimens, a columbium-1 percent zirconium tube coated with iron-titanate, had completed the prescribed 10,000 hours and the test was terminated. The testing of the remaining specimens is continuing. The preparation of the specimens and testing procedure are described in detail in a previous semiannual report, NASA - CR54268.

B. Iron-Titanate on Columbium-1 Percent Zirconium

With the addition of 3,750 hours of testing during the current report period, the columbium-1 percent zirconium tube coated with a 4-mil thick layer of iron-titanate completed 10,000 hours of testing in vacuum at 1700°F. The appearance of the specimen at 1700°F just prior to the end of the test is shown in Figure 1.

At the beginning of the test, the emittance was 0.88, but after 600 hours it decreased until, at 1800 hours, a value of 0.85 was reached. The emittance remained relatively stable for the remaining period with only a slight decrease from 0.85 to 0.84 during the final 2000 hours. The values for the 10,000-hour test are shown in Figure 2. The temperatures used for the final emittance values were obtained by using an optical pyrometer with a calibration traceable to the National Bureau of Standards.

During testing, the specimen was thermally cycled between 1700°F and room temperature 51 times with 22 of the cycles being made during the current report period. Thermal cycling produced no adverse effects on the specimen.

Throughout the test, a vacuum of 1×10^{-7} mm Hg or better was maintained as shown in Figure 2.

The appearance of the specimen after testing is shown in Figure 3. There were no apparent changes in the appearance of the coating other than a slight change in color. No spalling or cracking was evident. A photomicrograph of the coating is shown in Figure 4.

The specimen is now being subjected to spectrographic, X-ray diffraction, and vacuum fusion analysis, and microhardness testing. The results of the spectrographic analysis are presented in Table 1. As shown, essentially no change occurred.

TABLE 1

Spectrographic Analysis Results for
Iron-Titanate Coating on Columbium-
1 Percent Zirconium Tube

	<u>Impurity Content (Weight Percent)</u>						
	<u>Mn</u>	<u>Si</u>	<u>Mg</u>	<u>Al</u>	<u>Cr</u>	<u>Ni</u>	<u>Cu</u>
As received-powder	0.5	0.3	0.1	0.7	0.03	-	-
As sprayed	0.5	0.3	0.1	0.7	0.05	0.02	0.01
After 10,000 hrs at 1700°F	0.5	0.5	0.2	1.0	-	-	-

The small amounts of nickel and copper detected in the "as-sprayed" coating were probably picked up during the plasma-spraying of the coating. These elements have been detected in other coatings after plasma spraying.

X-ray diffraction analysis of the coating revealed the presence of no crystalline phases other than iron-titanate.

The vacuum fusion oxygen analysis made on a section of the tested columbium-1 percent zirconium tube indicated that about 0.33 percent oxygen was present after testing, whereas 0.03 percent was present before testing. This increase may be due in part to microscopic amounts of coating remaining on the tube surface after removal of the coating by scraping. A thorough microprobe analysis on the cross-section of the tube will be made. The results of this analysis, which will include an oxygen scan across the 10-mil wall section, will be reported in a later report.

Microhardness testing across the 10-mil coating cross-section indicated that the hardness of the substrate increased during testing for a depth of about 3 mils under both the inner and outer surfaces. The hardness midway between the surfaces was unchanged. These results are shown in Table 2 below:

TABLE 2

Microhardness Results for
Columbium-1 Percent Zirconium Tube
Coated With Iron-Titanate and Tested at
1700°F for 10,000 Hours

As Received		After 10,000 Hours at 1700°F	
Depth Below Outer Surface (Mils)	Diamond Pyramid Hardness (kg/mm ²)	Depth Below Outer Surface (Mils)	Diamond Pyramid Hardness (kg/mm ²)
1.5	146	0.5	191
2.5	146	1.5	196
4.0	144	2.5	170
5.5	142	4.0	141
6.75	146	5.0	144
8.0	146	6.5	168
		8.0	196

Note: Tube wall is 10 mils thick.

The increase in hardness at the inner surface of the tube (6 to 8 mils below the outer surface) is believed to be due to a formation of a thin oxide layer occurring during the plasma-spraying process. This oxide then diffused inward during the endurance test. The increase in hardness near the outer surface is believed to be due to some oxygen diffusing from the coating during the 10,000-hour test. Previous studies have indicated that no appreciable oxide formation occurs on the surface of the metal being sprayed during the plasma-spraying process. This is believed to be due to the preheat with argon, and the use of argon during the actual spraying process.

Ten thousand hours of testing an iron-titanate-coated columbium-1 percent zirconium specimen at 1700°F have demonstrated that the coating and substrate remain well bonded and that the coating provides a surface with a high, stable emittance. These characteristics indicate that this material combination is suitable for space radiator use at 1700°F, providing microprobe analyses to be made confirm that the materials are sufficiently compatible.

C. Calcium Titanate on AISI-310 Stainless Steel

An AISI-310 stainless steel tube coated with a 4-mil thick layer of

calcium titanate accrued a total of 10,600 hours by the end of the report period. Of these 10,600 hours, 4300 hours were accumulated during the report period.

To date, the emittance has been quite stable. Initially the emittance was 0.91, and, at present, it is 0.89. The emittance values and the vacuum maintained during the test are shown in Figure 5.

The specimen was thermally cycled 27 times between 1350°F and room temperature during the report period, bringing the total number of cycles to 51. No adverse effects on either adherence or emittance have been noted. The appearance of the specimen at 1350°F after 10,000 hours of testing is shown in Figure 6.

Based on over 10,000 hours of emittance testing and supplementary adherence testing, it appears that calcium titanate is a suitable coating material for AISI-310 stainless steel when used at 1350°F in vacuum.

D. Iron-Titanate on AISI-310 Stainless Steel

The testing of an AISI-310 stainless steel tube with a 4-mil thick iron-titanate coating was continued at 1350°F. During the report period, 4,300 hours were accrued for a total test time of 9,600 hours.

Throughout the test, the emittance has been very stable, having dropped only from 0.89 to 0.88 since the start of the test. The emittance values obtained are shown in Figure 7.

As shown in Figure 7, a vacuum of about 2×10^{-8} mm Hg has been maintained.

During the report period, the specimen was subjected to 27 additional thermal cycles between 1350°F and room temperature, bringing the total to 51 cycles. No adverse effects have resulted. The appearance of the specimen after 9,000 hours of testing is shown in Figure 8.

The emittance data obtained in conjunction with the results of vibration fatigue testing indicate that this material is suitable for use at 1350°F in vacuum.

E. Aluminum Oxide-Aluminum Titanate on Columbium-1 Percent Zirconium

A columbium-1 percent zirconium tube with a 4-mil thick coating of aluminum oxide-aluminum titanate is being tested at 1700°F. To date,

the specimen has accrued 5300 hours with 4300 hours of testing being conducted during the report period.

At the start of the test, the emittance was 0.87, but it has decreased steadily. By end of the report period, the emittance had dropped to 0.82. Projected emittance values are unsatisfactory, and, if the emittance continues to drop, the test will be terminated before 10,000 hours are accumulated. The emittance values recorded for this specimen and the vacuum maintained are shown in Figure 9. The specimen has been subjected to a total of 36 thermal cycles with 27 of the cycles being conducted during the report period. The appearance of the specimen after 4700 hours of testing is shown in Figure 10.

IV. BERYLLIUM STUDIES

A. Introduction

A study is being conducted to determine the feasibility of using coated beryllium for radiator applications. Specimens are prepared by plasma-arc spraying high emittance materials onto beryllium samples. The specimens are then aged at 800°F or 1400°F in vacuum for periods of 100, 500, and 1000 hours. Before and after aging, the specimens are thoroughly examined to determine any changes in the coating or substrate. The examination includes chemical analysis of the substrate and coating and determination of the substrate microstructure, substrate hardness, and coating-substrate bond strength. The emittance properties of the coating materials when applied to beryllium are being determined with separate specimens.

The coatings initially selected for evaluation are iron-titanate and calcium titanate. Both of these materials have exhibited high emittance and good bond strengths for periods of at least 10,000 hours on other metal substrates as indicated in Section III of this report.

B. Aging Tests

1. Apparatus

In order to accomplish the work required for the beryllium phase of the program, it was necessary to increase the size of the emittance facility. Two rooms were added, one for specimen preparation, and one to house the test equipment. The room used for specimen preparation was constructed with smooth interior walls and a tile floor to conform to health and safety engineering requirements.

Two furnaces were constructed for aging the specimens. Each is a general purpose box-type bench furnace capable of maintaining temperatures up to 2300°F (see Figure 11). Provision is made for installing a hot-wall vacuum chamber 6 inches in diameter and 22 inches long. Three thermocouples are mounted along the length of the chamber exterior to monitor the furnace temperature.

The specimens are mounted in a stainless steel sample holder specially constructed for the purpose. Four Chromel-Alumel thermocouples are welded to the sample holder to provide the temperature profile of the test area.

A titanium sublimation pump was constructed for use with either furnace assembly to provide a larger pumping speed than is available with an ion gettering pump if required.

All basic furnace controls and instrumentation read-out units are grouped in a console. Included are temperature controllers for the furnaces, power supplies for the ion gettering pumps, an ionization gage control unit, a thermocouple gage control unit for use during bakeout, a switching arrangement and a slidewire millivolt potentiometer for thermocouple output measurement, and the control unit for the titanium sublimation pump. The console is shown in Figure 12.

2. Specimen Preparation

The specimens being used for the aging tests are coated beryllium plates measuring 3 inches by 3 inches and 0.10 inch thick. The beryllium was procured from the Beryllium Corporation and was designated as high purity IS-2 ingot sheet. Spectrographic analysis of the beryllium sheet, as received, indicated the presence of the following impurities: 0.06% Si, 0.05% Fe, 0.02% Ni, 0.01% Ti, 0.01% Mn, and traces of Cr, Cu, Mg, Pb, Au, and Sn. Chemical analysis showed 0.8% BeO to be present. Microhardness testing indicated that the surfaces of the plates were slightly harder than the material away from the surfaces (see Table 3), apparently as a result of cold working during fabrication.

TABLE 3

Microhardness Results for
Beryllium Plates (as Received)
Used in Aging Studies

Depth Below Outer Surface (Mils)	Diamond Pyramid Hardness (kg/mm ²)
0.5	227
1.5	227
3.0	219
4.5	219
6.0	201
8.0	201
10.0	201
12.5	201
50	202

The specimens are prepared for coating by grit blasting with 60 mesh silicon carbide at 80 psi to a roughness height of about 80 microinches AA. Coatings are applied by plasma-arc spraying, using argon for both the arc gas and the carrier gas.

During the report period, specimens were prepared with 4-mil thick coatings of iron-titanate and calcium-titanate. The iron-titanate powder used was coarser and more uniform than that used previously to prepare emittance specimens. Use of a coarser powder eliminated powder compaction in the spray gun hopper, thus ensuring a steady flow to the gun. The particle size distribution of the powder is shown in Table 4.

TABLE 4

Particle Size Distribution of
Iron-Titanate Powder
Used for Beryllium Studies

<u>Particle Size</u> <u>(Microns)</u>	<u>Cumulative</u> <u>Weight Percent</u>
72	37
66	44
60	54
52	63
42	74
33	82
26	88
21	90
17	94
9.5	99
4.1	100

Spectrographic analyses were conducted for both powders. Traces of Si and Mg were found in the calcium-titanate powder. The iron-titanate powder was found to contain 0.6% Mn, 0.4% Si, 0.4% Al, and 0.3% Mg.

3. Test Procedure

Aging is conducted in one of the two furnaces described previously. The coated beryllium plates are installed in the sample holder and the holder assembly is inserted into the vacuum chamber. The vacuum chamber is then sealed and baked out for six hours at 500°F. Heat during the bakeout period is supplied by the furnace, by a heated wire on the manifold, and by a bakeout mantle installed on the ion gettering pump. A 400-liter-per-second oil diffusion pump backed by a liquid nitrogen cold trap and mechanical pump is used to evacuate the system. The pressure during the roughing and bakeout period is measured by a thermocouple gage located in the vacuum chamber manifold. After bakeout, the system is cooled, the ion gettering pump is started, and the roughing pump is valved out of the system. The vacuum chamber temperature is then raised to the desired aging temperature.

The oven temperature is held constant by a controlling pyrometer, and the sample holder temperatures are measured with a millivolt slide-wire potentiometer. The chamber pressure is measured with an ionization gage and by measuring the ion gettering pump current and using a calibration curve of pump current vs chamber pressure. At the end of the required aging time, the furnace is shut down, the chamber is cooled and filled with dry nitrogen to atmospheric pressure, and then the specimens are removed for evaluation.

4. Test Results

By the end of the report period, aging tests had been conducted on calcium-titanate-coated specimens for 100 and 500 hours and on iron-titanate-coated specimens for 100 hours at 800°F.

a. Calcium-Titanate-Coated Beryllium Specimens

Three calcium-titanate-coated beryllium specimens were aged at 800°F for 100 hours in a vacuum of 1×10^{-7} mm Hg or better. Visual examination of the specimens after aging revealed no changes in their appearance other than a slight darkening of the coating. The texture was unchanged, and there was no evidence of spalling or cracking of the coating. The appearance of the specimens before and after aging is shown in Figure 13. The specimens were weighed before and after aging, and no significant weight change was found. The weights of the specimens before and after testing are shown in Table 5.

TABLE 5

Weights of Calcium-Titanate-Coated Beryllium
Specimens Before and After Testing at 800°F

<u>Specimen</u>	<u>Aging Time (hr)</u>	<u>Weight (gm)</u>	
		<u>Before Test</u>	<u>After Test</u>
1	100	30.482	30.481
2	100	30.493	30.489
3	100	30.980	30.978
4	500	29.744	29.740
5	500	30.008	30.004
6	500	29.816	29.810

One of the three specimens is being subjected to metallurgical and chemical examination. Preliminary results indicate that no diffusion

occurred between the coating and the substrate. One indication of this is the results of microhardness testing. A diamond pyramid hardness value of 201 kg/mm^2 was obtained at depths of 0.2, 25, and 50 mils below the coating-substrate interface. Spectrographic analyses were made on scrapings from the specimen. Comparison of these results with those of the analysis performed prior to aging reveals that no significant changes occurred (see Table 6).

TABLE 6

Spectrographic Analysis Results for
Calcium-Titanate and Iron-Titanate
Applied to Beryllium Substrates

<u>Material and History</u>	<u>Impurities</u>
<u>Calcium-Titanate</u>	
As received	Traces of Si, Mg
As sprayed	0.02% Si, 0.01% Cu, traces of Ni, Al, Fe, Mg, Mn
100 hours at 800°F	0.01% Si, traces of Al, Fe, Mg, Mn
500 hours at 800°F	0.01% Si, traces of Al, Fe, Mg, Mn
<u>Iron-Titanate</u>	
As received	0.6% Mn, 0.4% Si, 0.4% Al, 0.3% Mg
As sprayed	0.5% Mn, 0.4% Si, 0.5% Al, 0.2% Mg, 0.01% Cu, trace of Ni
100 hours at 800°F	0.6% Mn, 0.5% Si, 0.7% Al, 0.3% Mg, traces of Cr, Cu

Three additional calcium-titanate-coated beryllium specimens were aged for 500 hours at 800°F in a vacuum of 1×10^{-7} mm Hg or better. The results were similar to those for specimens aged 100 hours. Visual examination after aging revealed no changes other than a slight darkening of the coating, and the change in weight was insignificant. The appearance of one of the aged specimens is shown in

Figure 13. As shown, the color change was about the same as that of the specimen aged 100 hours. Microhardness testing revealed a uniform hardness in the beryllium as shown in Table 7.

TABLE 7

Microhardness Results for
Beryllium Plate Coated with
Calcium-Titanate and Aged for
500 Hours at 800°F

<u>Depth Below Interface (Mils)</u>	<u>Diamond Pyramid Hardness (kg/mm²)</u>
0.5	177
1.75	179
3.0	179
4.5	179
5.0	177

The results of spectrographic analyses of the coating after aging are shown in Table 6. No appreciable change in the coating impurities occurred. As shown in Table 5, no significant change in weight occurred.

The aging tests conducted to date on calcium-titanate-coated beryllium specimens demonstrate that calcium-titanate is chemically compatible with beryllium and will remain bonded to it for periods of at least 500 hours at 800°F in vacuum.

b. Iron-Titanate-Coated Beryllium Specimens

Three beryllium specimens coated with iron titanate were aged for 100 hours at 800°F in a vacuum of 1×10^{-7} mm Hg or better.

Visual examination of the specimens after aging revealed no changes in color or texture, and no spalling or cracking were evident. The change in weight during aging (see Table 8) was considered to be insignificant. The appearance of one of the specimens before and after aging is shown in Figure 14.

TABLE 8

Weights of Iron-Titanate-Coated Beryllium Specimens
Before and After Testing at 800°F for 100 Hours

<u>Specimen</u>	<u>Weight (gm)</u>	
	<u>Before Test</u>	<u>After Test</u>
1	30.356	30.350
2	30.451	30.448
3	29.929	29.924

Microhardness testing revealed that the substrate material close to the interface was somewhat harder than that towards the middle of the substrate thickness (see Table 9). These results may possibly indicate that some diffusion occurred between the coating and the substrate during aging. If the results for samples aged for 500 and 1,000 hours are similar, microprobe analyses will be performed to determine the cause of the hardness change.

TABLE 9

Microhardness Results for
Beryllium Plate Coated
with Iron-Titanate

<u>Depth Below Interface (Mils)</u>	<u>Diamond Pyramid Hardness (kg/mm²)</u>
0.5	227
1.5	182
2.5	188
3.5	185
50	185

Spectrographic analyses of the coating before and after aging revealed no change in the amount of elemental impurities. These results are presented in Table 6.

On the basis of these results, it appears that iron-titanate is chemically compatible with beryllium and that it maintains good bond integrity to beryllium for at least 100 hours at 800°F in vacuum.

C. Emittance Tests

1. Introduction

As a part of the beryllium studies, beryllium strips are coated with the same materials used for the aging tests, and the resulting specimens are tested for emittance stability at temperatures between 800 and 1400°F. The beryllium strips used are 9 inches long, 0.4 inch wide, and 0.010 inch thick. Four-mil-thick coatings are applied by plasma-arc spraying using the same procedure as that used for the aging specimens. The total hemispherical emittance tests are conducted in the short-term endurance rig described in Pratt & Whitney Aircraft Report PWA-2518 (NASA CR-54268).

2. Test Results

One emittance test was conducted during the report period. The specimen was a beryllium strip coated with a 4-mil-thick layer of iron-titanate. Emittance values were obtained at 100°F intervals between 800°F and 1100°F over a period of 670 hours. The emittance values were all between 0.89 and 0.90, as shown in Figure 15.

After testing at 1100°F, one of the thermocouples failed, and it was necessary to interrupt the test to reinstrument the specimen. Emittance testing will be continued during the next report period up to 1400°F.

The appearance of the specimen after testing at 1100°F is shown in Figure 16. The specimen appeared to be unchanged by this portion of the test.

V. CONCLUSIONS

On the basis of at least 10,000 hours of emittance testing and related adherence testing, it is concluded that iron-titanate on either AISI-310 stainless steel or on columbium-1 percent zirconium and calcium-titanate on AISI-310 stainless steel are suitable for space radiator use at 1350°F (with a stainless steel substrate) or 1700°F (with a columbium-1 percent zirconium substrate). All of these material combinations are unaffected by thermal cycling and have demonstrated good adherence properties during fatigue testing. Iron-titanate applied to columbium-1 percent zirconium has an emittance between 0.88 and 0.84. When applied to AISI-310 stainless steel, it has an emittance between 0.89 and 0.88. Calcium titanate on AISI-310 stainless steel has an emittance between 0.91 and 0.89.

Aluminum oxide-aluminum titanate on columbium-1 percent zirconium does not appear to be suitable for space radiator applications at 1700°F. After 5300 hours of testing at this temperature, the emittance has dropped from 0.87 to 0.82.

The aging of beryllium plates coated with calcium titanate for 500 hours at 800°F does not appear to produce any significant changes in either the coating or the substrate. Aging beryllium plates coated with iron titanate for 100 hours at 800°F also produces no significant changes. The emittance of a beryllium strip coated with iron-titanate was found to be between 0.89 and 0.90 over a temperature range from 800°F to 1100°F during 670 hours of testing.

VI. FUTURE WORK

The remaining long-term total hemispherical emittance tests being conducted on coated AISI-310 stainless steel and columbium-1 percent zirconium tubes will be continued up to at least 10,000 hours if the emittance remains stable. Those with AISI-310 stainless steel substrates may be continued for a longer period. The test of aluminum oxide-aluminum titanate on columbium-1 percent zirconium will be terminated before 10,000 hours are reached if the emittance continues to decrease.

The beryllium studies will be continued to evaluate the compatibility, emittance stability, and adherence of iron titanate and calcium titanate to beryllium. It is expected that a third coating material will be introduced during the next six-month period.

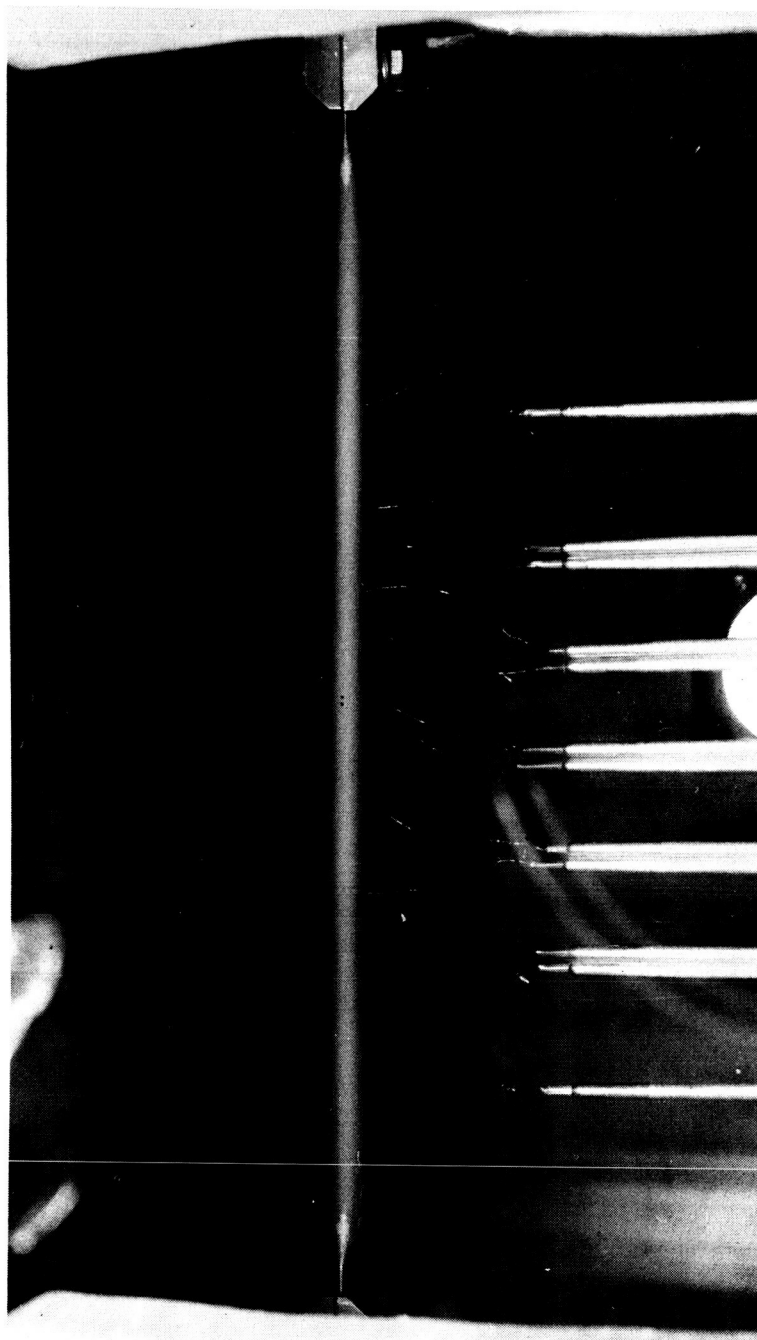


Figure 1 - Iron-Titanate-Coated Columbium-1 Percent Zirconium Specimen at 1700°F After 10,000 Hours of Testing

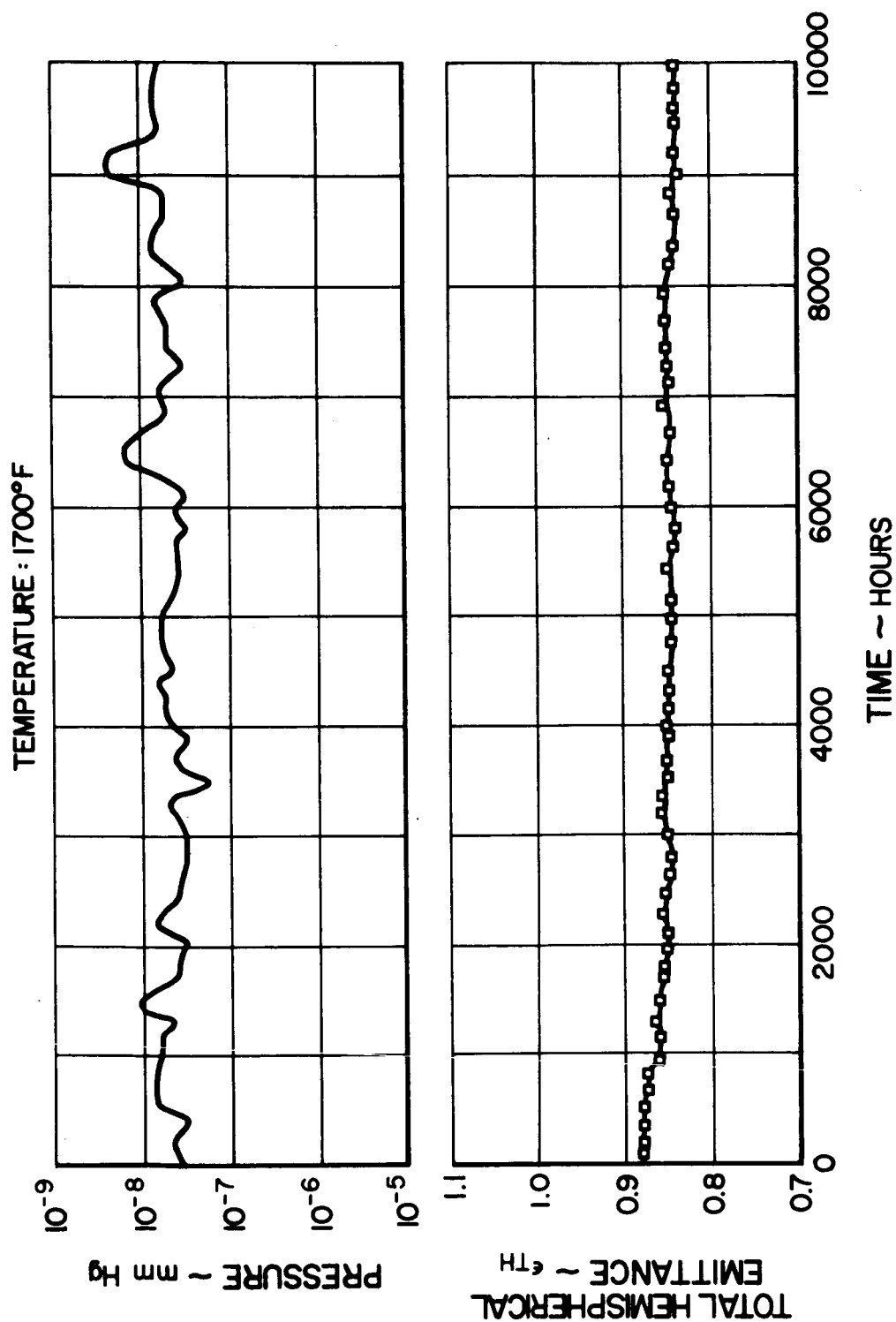


Figure 2 - Pressure and Total Hemispherical Emittance for Iron-Titanate-Coated Columbium-1 Percent Zirconium Specimen

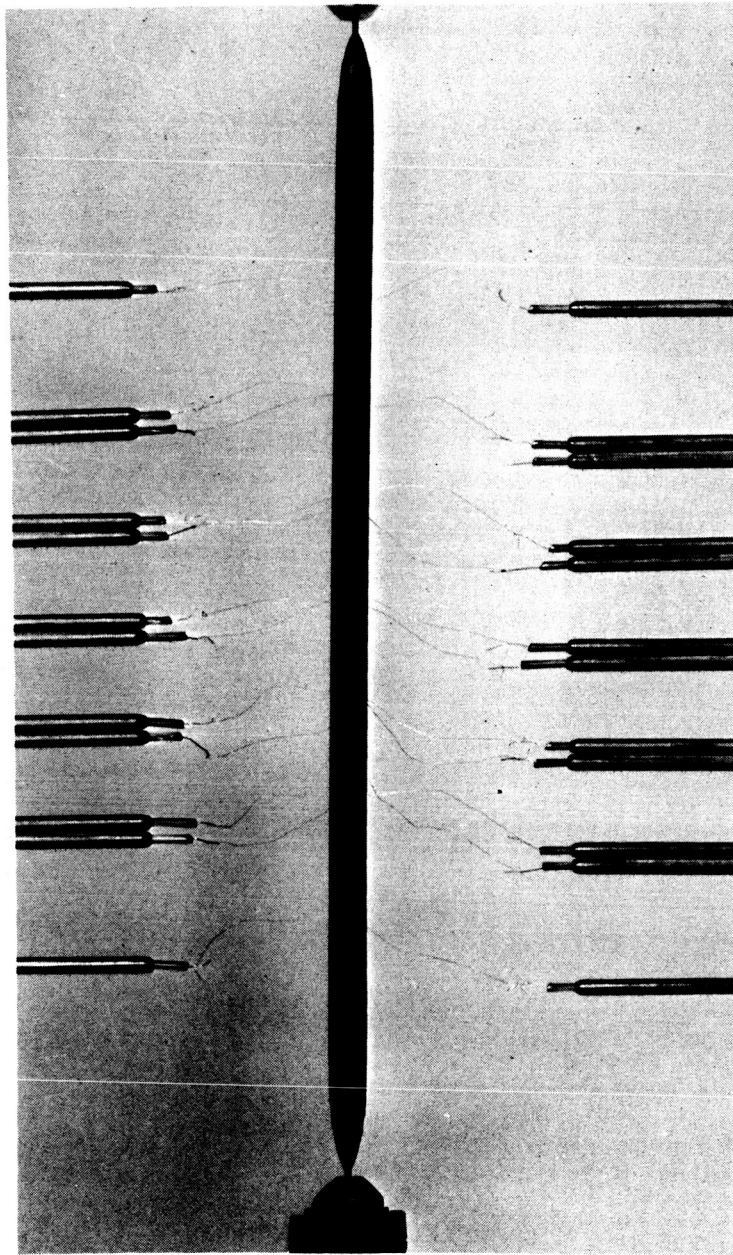


Figure 3 - Iron-Titanate-Coated Columbium-1 Percent Zirconium Specimen After 10,000 Hours of Testing at 1700°F

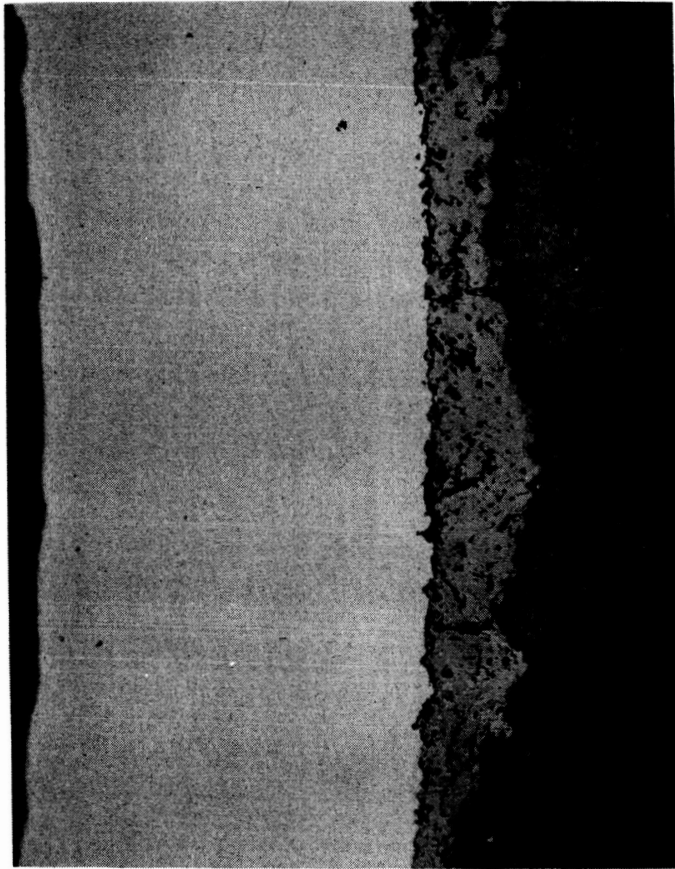


Figure 4 - Photomicrograph of Iron-Titanate on Columbium-1
Percent Zirconium After 10,000 Hours of Testing
at 1700°F

Mag: 200X

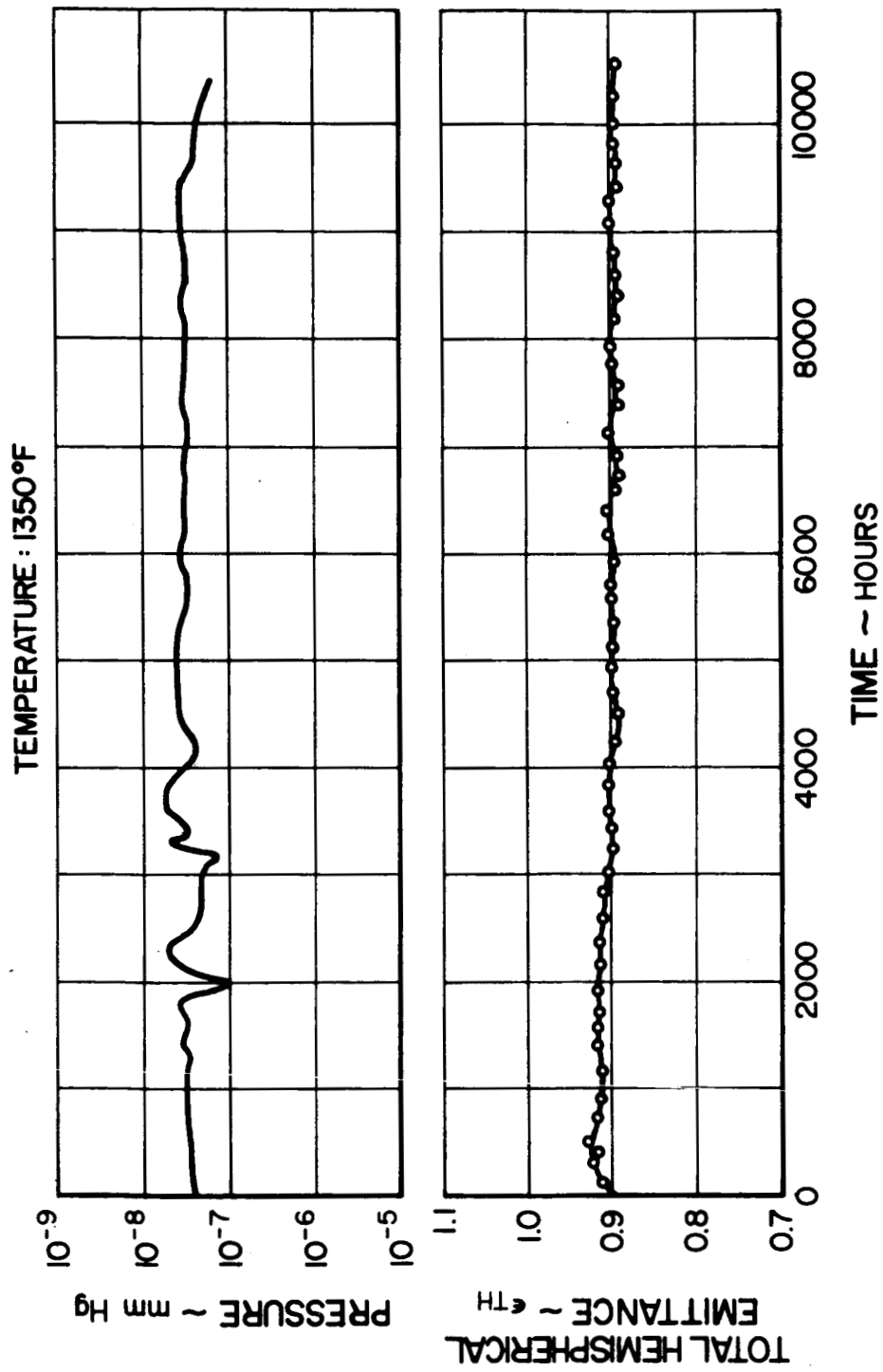


Figure 5 - Pressure and Total Hemispherical Emittance for Calcium-Titanate-Coated AISI-310 Stainless Steel Specimen

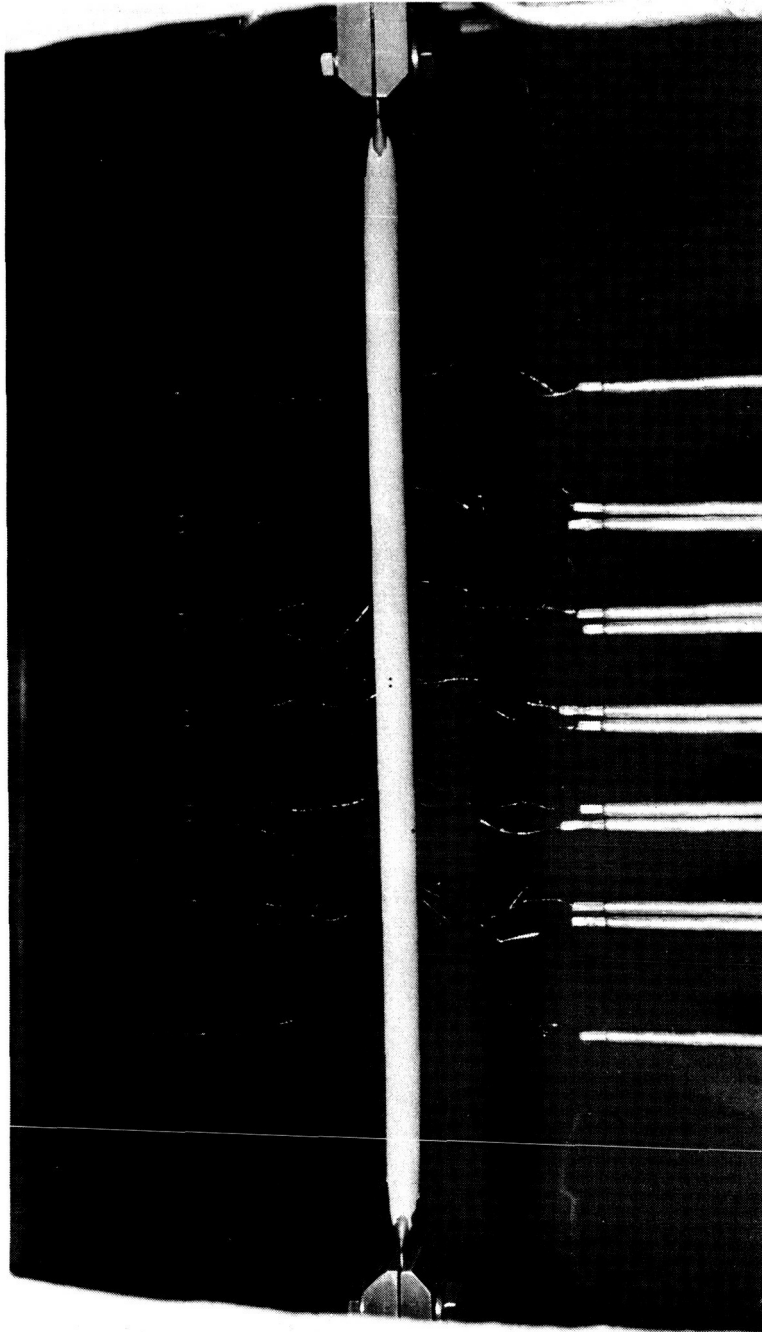


Figure 6 - Calcium-Titanate-Coated AISI-310 Stainless Steel Specimen After 10,000 Hours of Testing at 1350°F

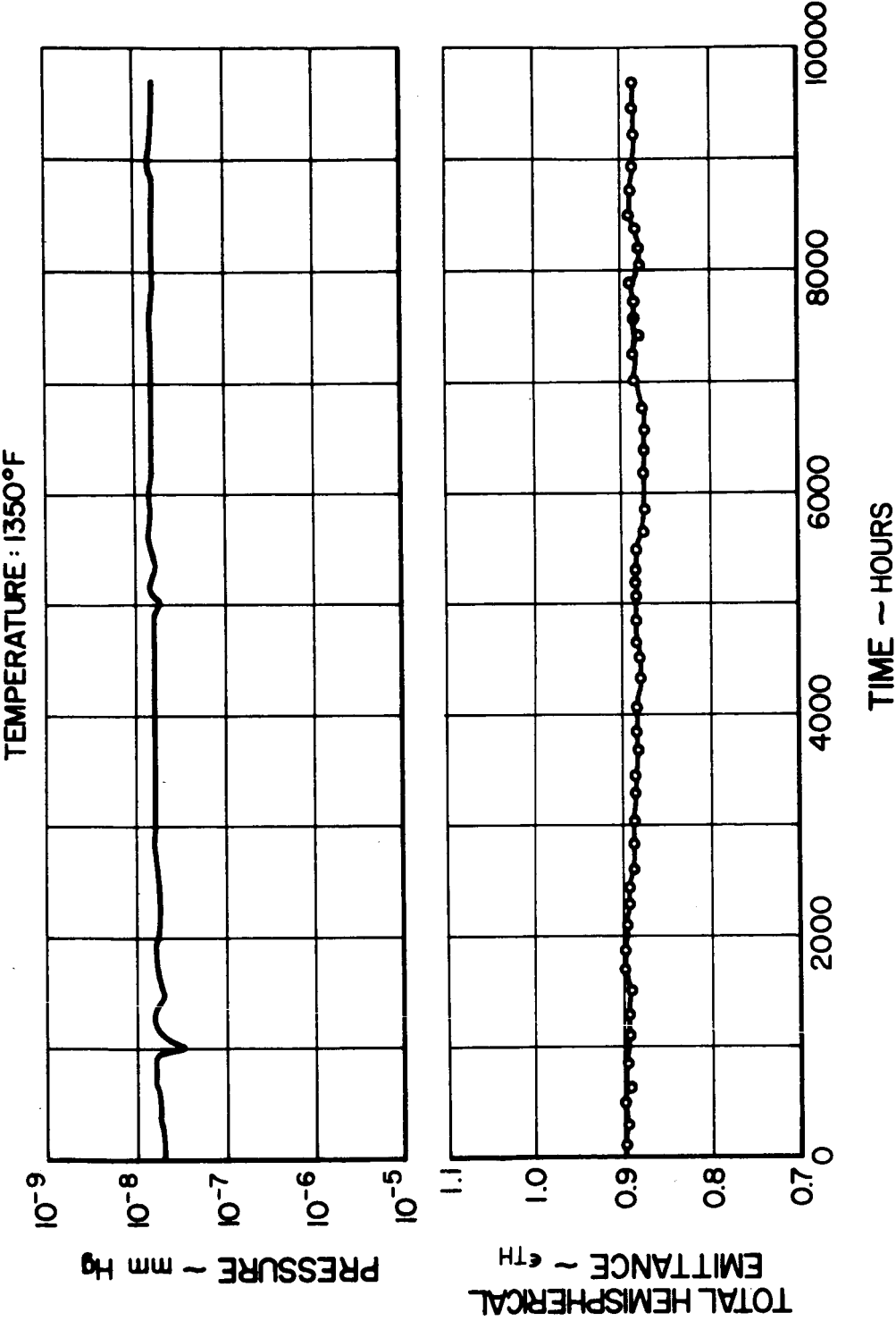


Figure 7 - Pressure and Total Hemispherical Emittance for Iron-Titanate on AISI-310 Stainless Steel

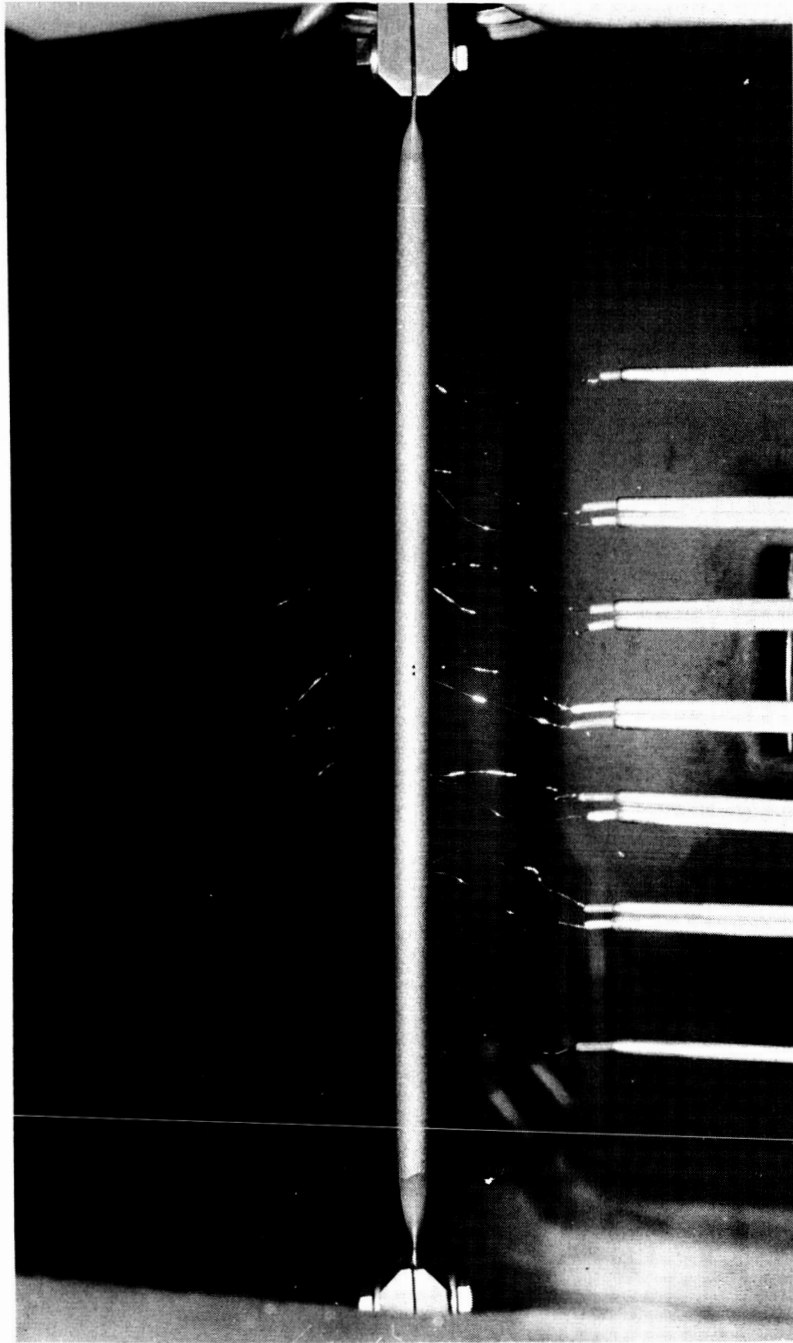


Figure 8 - Iron-Titanate-Coated AISI-310 Stainless Steel Specimen
After 9,000 Hours of Testing at 1350°F

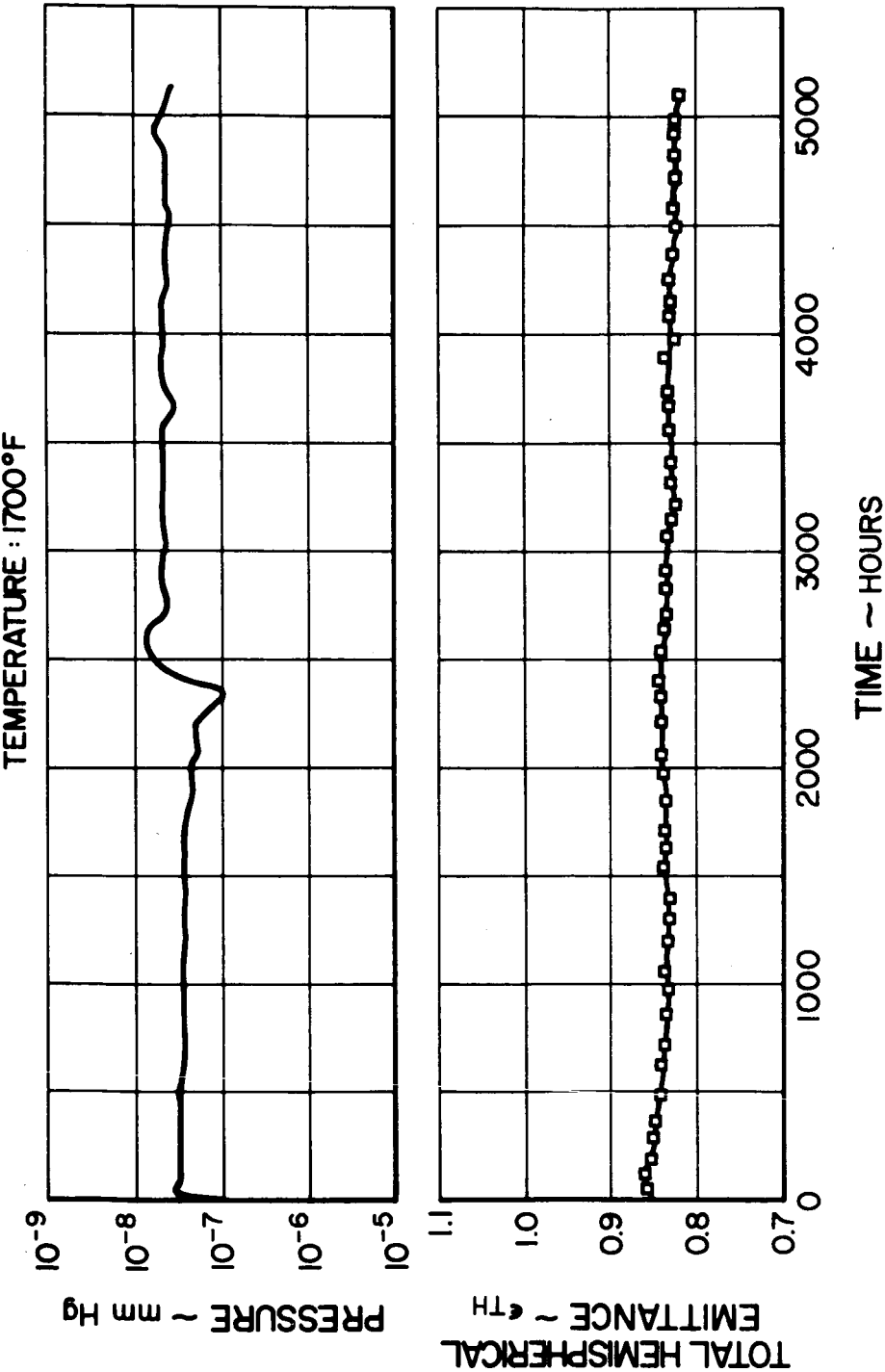


Figure 9 - Pressure and Total Hemispherical Emittance for
Aluminum Oxide-Aluminum Titanate-Coated
Columbium-1 Percent Zirconium



Figure 10 - Aluminum Oxide-Aluminum Titanate-Coated
Columbium-1 Percent Zirconium Specimen After
4,823 Hours of Testing at 1700°F

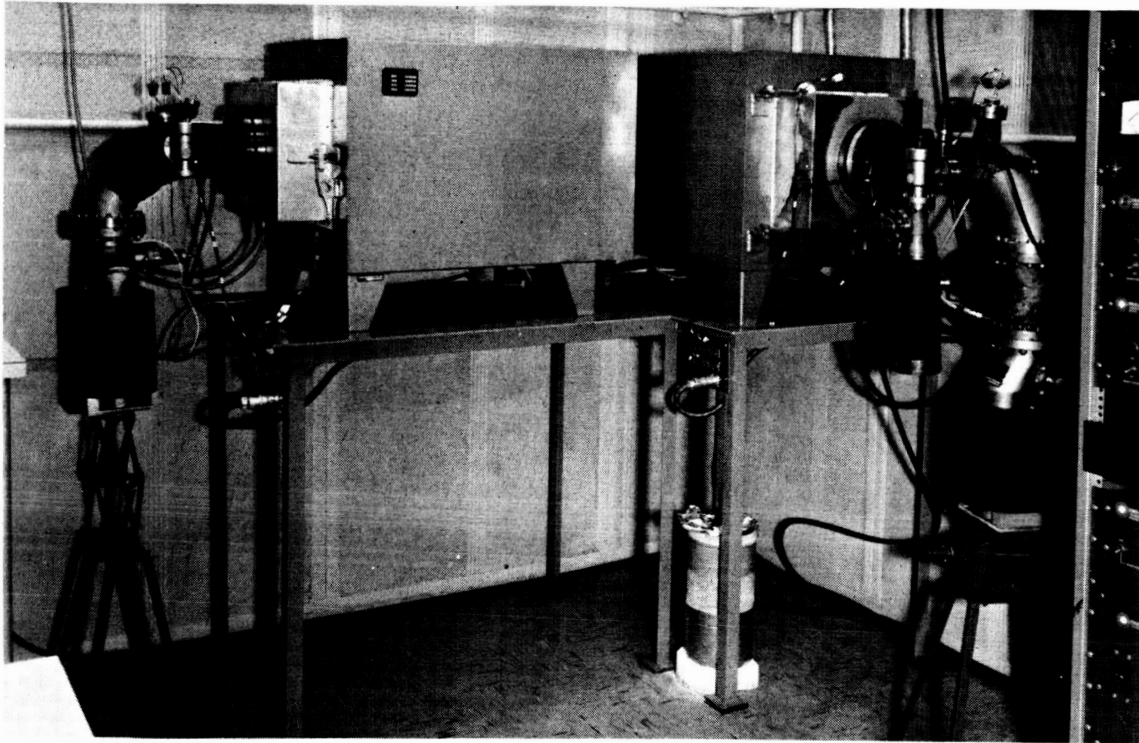


Figure 11 - Vacuum Aging Furnaces Used for Beryllium Studies



Figure 12 - Control Console for Beryllium Aging Studies

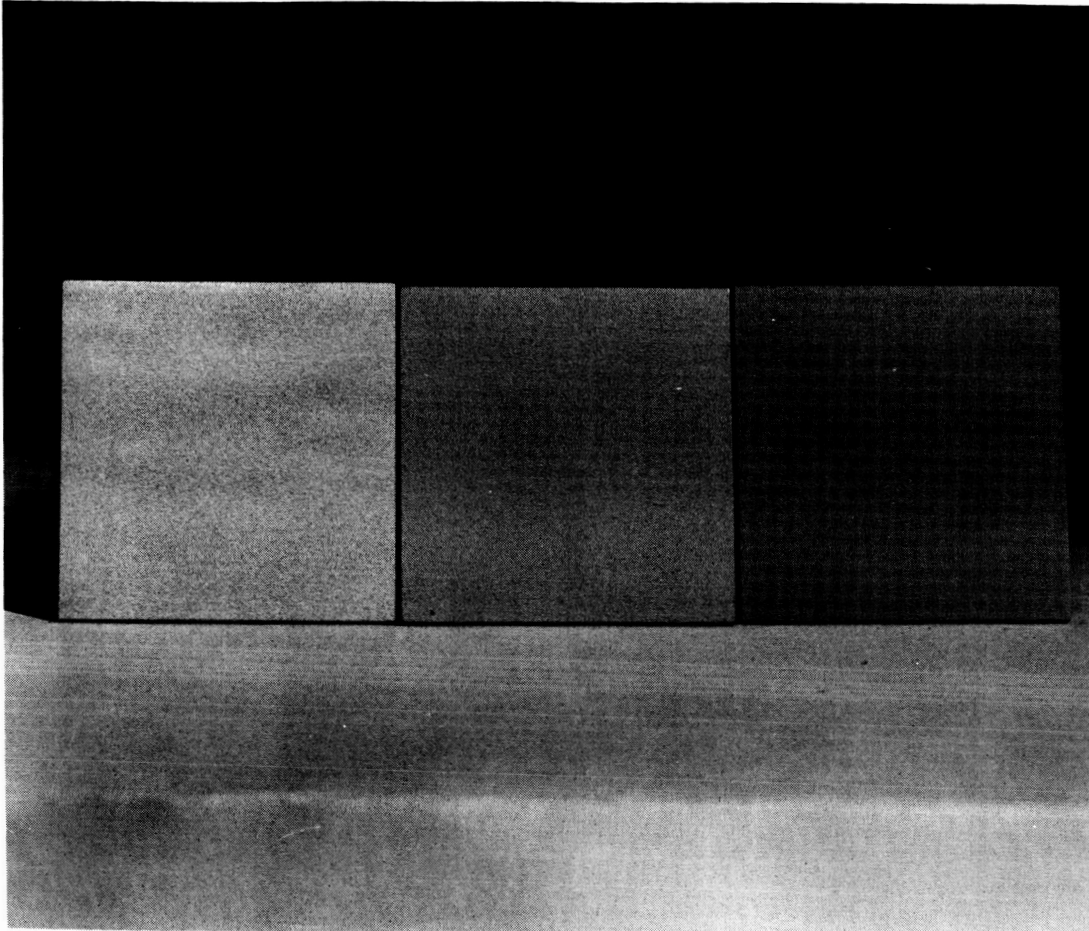


Figure 13 - Appearance of Calcium-Titanate-Coated Beryllium Specimen Before Aging (Left), After Aging for 100 Hours at 800°F (Center), and After Aging for 500 Hours at 800°F (Right)

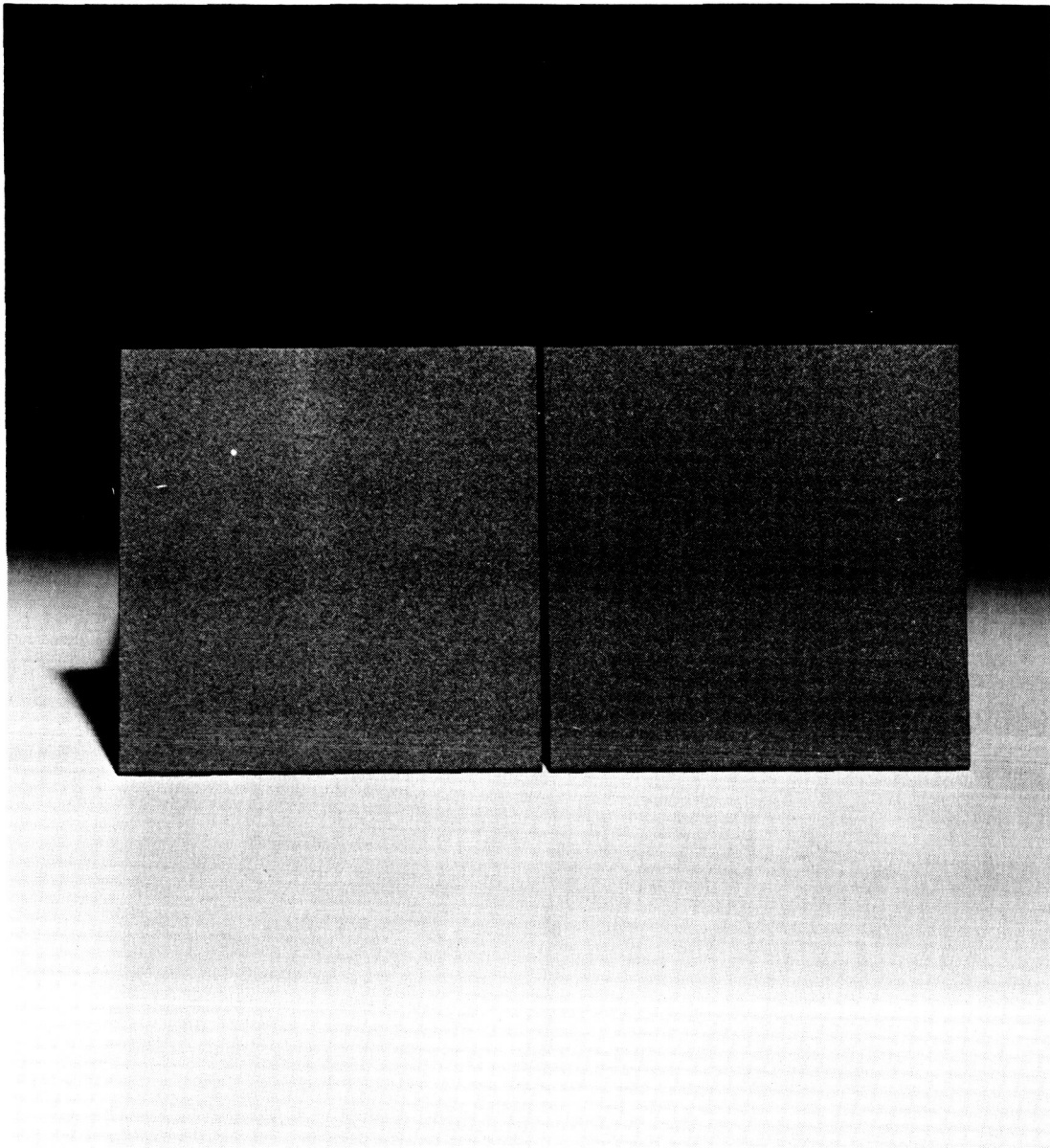


Figure 14 - Appearance of Iron-Titanate-Coated Beryllium Specimen Before Aging (Left) and After Aging for 100 Hours at 800°F (Right)

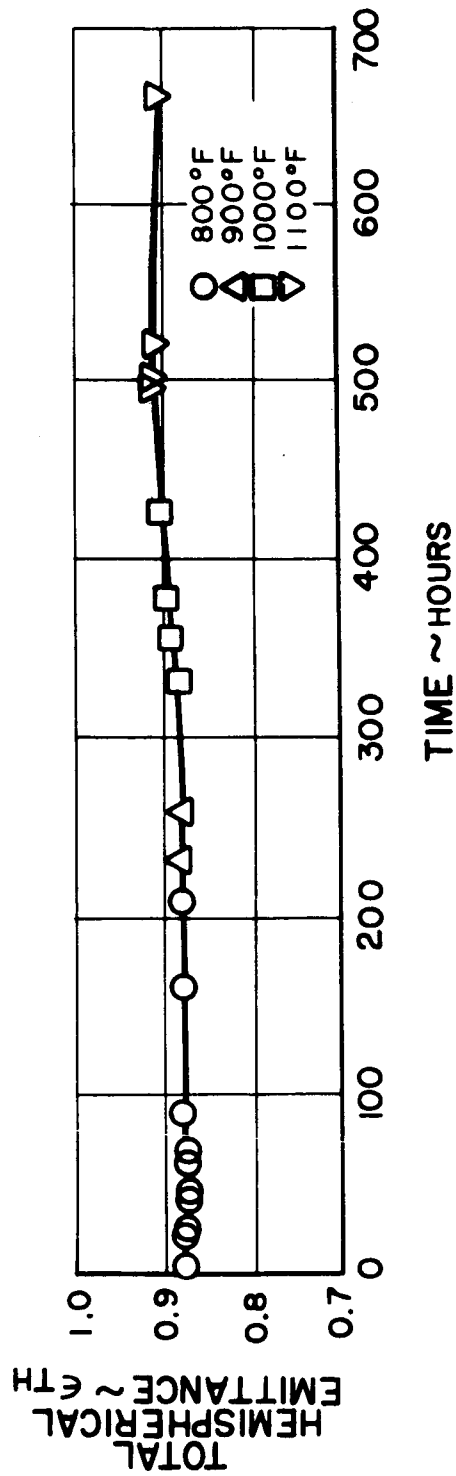


Figure 15 - Total Hemispherical Emittance of Iron-Titanate-Coated Beryllium

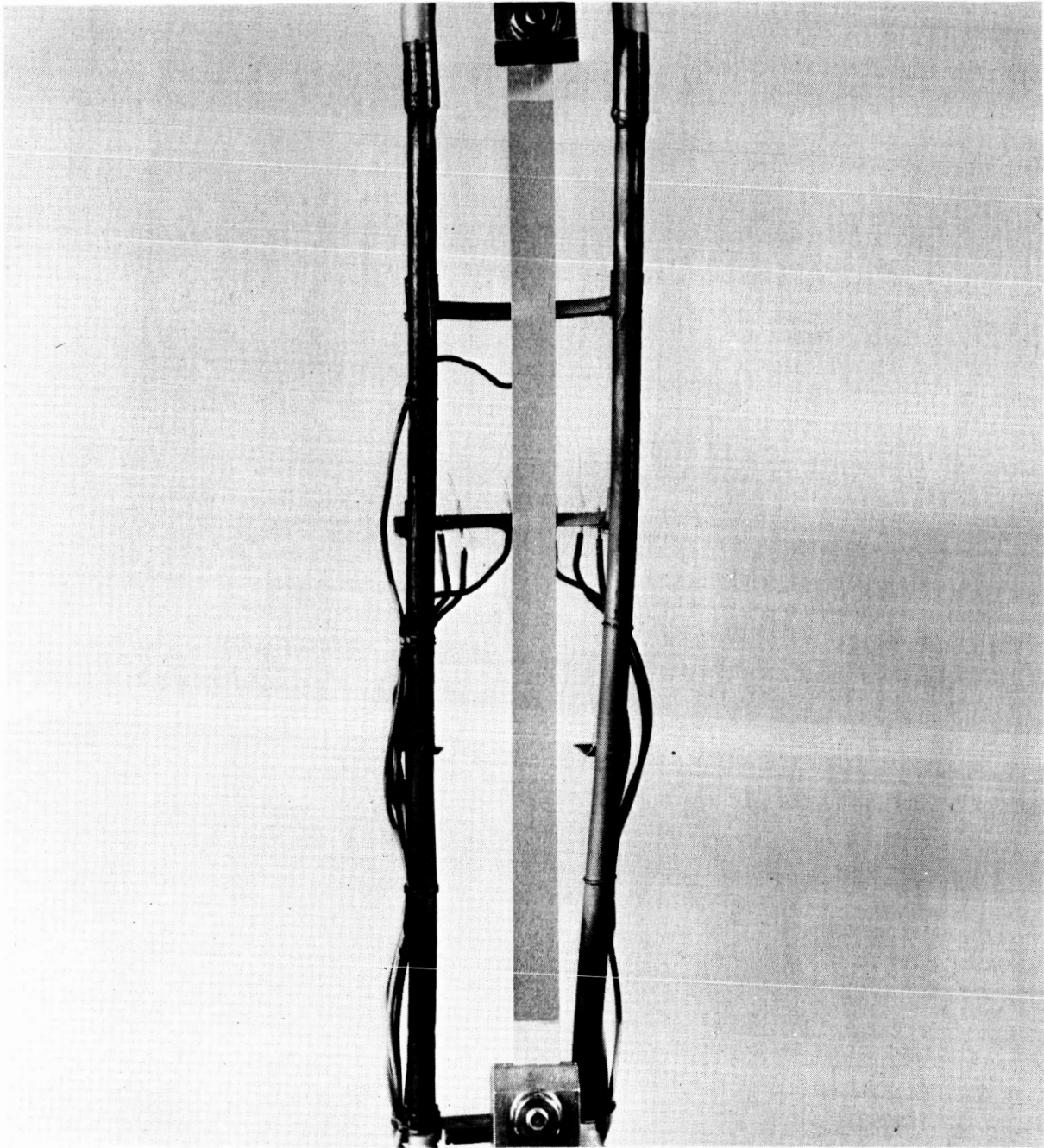


Figure 16 - Iron-Titanate-Coated Beryllium Specimen After Emittance Testing at Temperatures from 800°F to 1100°F